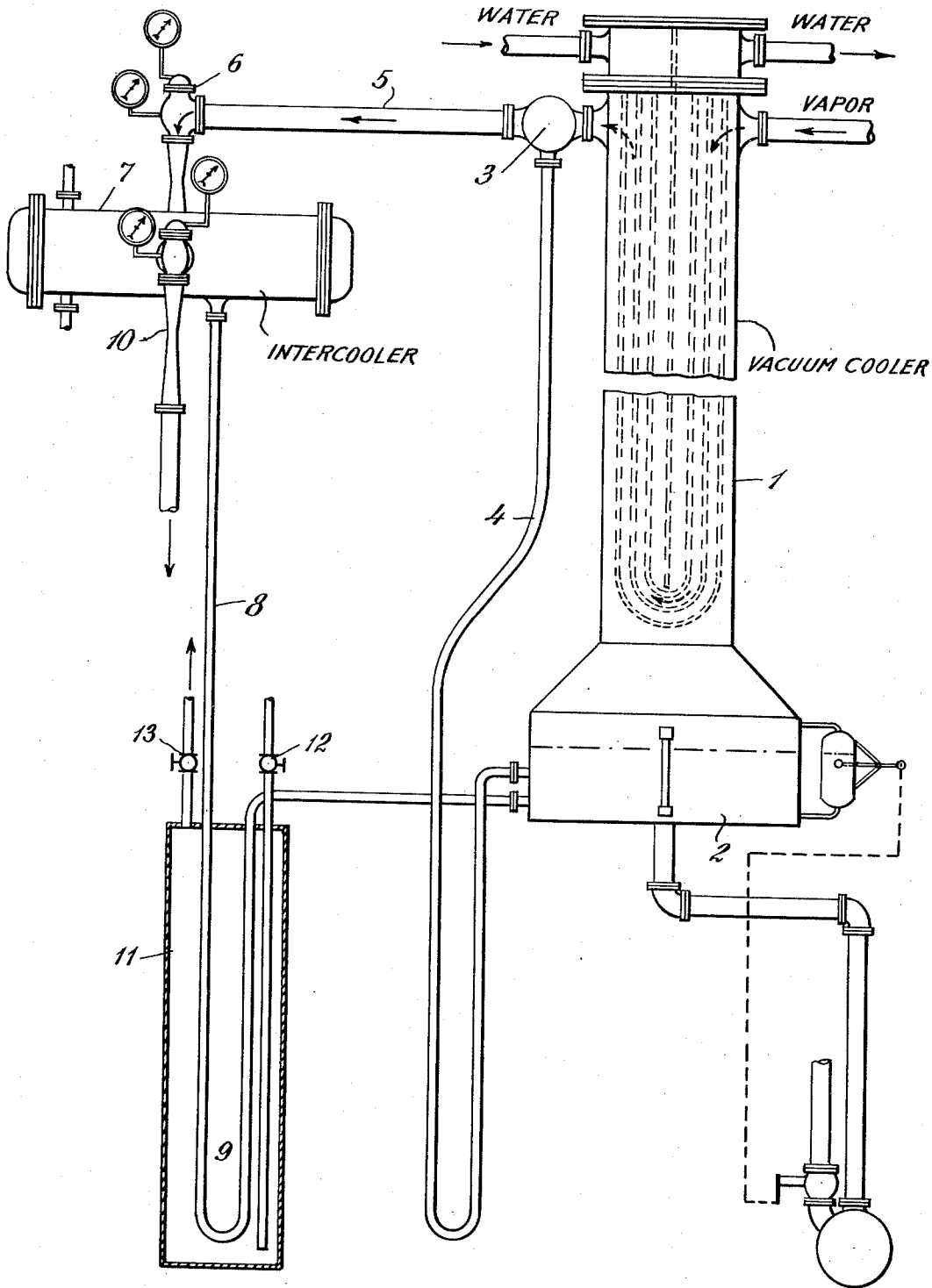
April 10, 1934. C. E. TARTE ET AL 1,954,371

METHOD AND APPARATUS FOR DISTILLATION UNDER VACUUM

Filed Aug. 19, 1933



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Patented Apr. 10, 1934 1,954,371

UNITED STATES PATENT OFFICE

1,954,371

METHOD AND APPARATUS FOR DISTILLATION UNDER VACUUM

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Application August 19, 1933, Serial No. 685,882

3 Claims. (Cl. 202—69)

This invention has to do with improvements in methods and apparatus for distillation and fractionation of oils and other materials under high vacuum. Specifically it has to do with methods and means for controlling communication between separate portions of the system under different degrees of vacuum.

In the operation of distillation and fractionation systems under high vacuum, it is the usual practice to pass the overhead vapors from the fractionating step into a condenser of the surface type. These vapors will contain not only condensable produce, but will contain light vapors of the same nature incondensable at the temperature and pressure existing in the surface condenser, and especially if steam has been used in the distillation, may contain a considerable amount of air. The vapors normally incondensable in the surface container are removed from the surface condenser both to preserve the vacuum and to allow efficient working of the surface condenser heat exchange surface. In high vacuum installations they are usually removed by multi-stage steam jet "air pumps". These devices consist of two or more nozzles, coupled in series, whereby a jet of steam entrains the gases, and compresses them to a higher pressure, followed by sufficient repetitions of the operation to enable discharge into the atmosphere or into a body at atmospheric or higher pressure. In order to allow economy in the use of steam, as the steam from each jet would otherwise be just so much gas to be moved by the jet next in series, it is usual to provide smaller surface condensers between jets to condense a substantial amount of the steam. In the usual installation, the vacuum device consists of a two stage steam jet air pump with a single intercooler.

The condensate in this intercooler, because of entrainment, carries a measurable amount of the product which should have been condensed in the first surface condenser, and so is returned to the liquid collection space in the first or main condenser. This return is carried out by means of a drain pipe leading from the intercooler to the surface condenser, having a loop in it to prevent vapor bypassing from the intercooler to the vacuum condenser which is at a lower pressure. When operating upon materials of higher degrees of volatility, we have found these seal loops in the drain leg to be ineffective. Theoretically they provide a column of liquid of sufficient height to counterbalance the difference in pressure. The liquid is at the saturation temperature corresponding to the pressure existing in the intercooler. That pressure is above the pressure existing in the vacuum cooler. Therefore the vapor pressure of the liquid in the drain seal loop is greater than the pressure in the vacuum condenser to which it connects. This causes vaporization of the liquid on the vacuum condenser side of the loop, and the unbalanced pressure will then clear the loop and the intercooler and vacuum condenser will be in open communication for a time until fresh condensate collects. Thus the "seal" operates in a manner to defeat its purported object. This continual fluctuation of vacuum, even though it be minor in amount, has far reaching effects. It disturbs the equilibrium conditions of disillation, leading to poorer stripping of light fractions; it is apt to cause surges of pressure in the tower resulting in poor fractionation; it renders the control of reflux difficult; and renders inaccurate the end point control of the distillate made.

It has therefore been an object of this invention to promote the accuracy and ease of control of distillation and fractionation operations carried out under high vacuum by providing means whereby the transfer of liquid mediums from one pressure stage to another can be carried out with positive assurance that there will be no pressure by-pass.

We have found that if the drain seal loop be separately cooled to a temperature below that at which its vapor pressure equals the pressure in the vacuum condenser this object may be accomplished.

This is a very different thing from the obvious one of cooling to this degree in the intercooler. The vapors and condensate are intermixed in the intercooler, and it is well known that in the presence of incondensable vapors and fixed gases, the coefficient of heat transfer is small. Sufficient surface to cause this cooling in the intercooler would be well nigh prohibitive. Not only does the lack of good heat transfer enter, but if the desired condensate were cooled to the desired temperature it would also be necessary to handle a large amount of undesired condensate at this point. But the amount of desired condensate is comparatively small, and after its removal from the intercooler it can be cheaply cooled in a small and efficiently operated cooler. This problem arises to an annoying magnitude when the distillation is one in which a major portion of the overhead product is light fractions, and in which the temperatures are comparatively low. In heavy oils, atmospheric cooling has been sufficient to suppress it. But in handling light

oils, atmospheric cooling is neither sufficiently great nor sufficiently reliable.

In order that our invention may be understood, reference is made to the drawing attached to and made a part of this specification. This drawing is a diagrammatic view of the condensers and vacuum devices upon a vacuum still. 1 is the vacuum condenser, with a liquid collection space 2 at the bottom, from which liquid is withdrawn in the usual manner. Fixed gases and vapors uncondensed in the vacuum condenser pass into precooler 3, and the small amount of condensate produced therein is returned through drain leg 4 to liquid space 2. Gases from precooler 3 are drawn through line 5 by steam jet 6 and forced into intercooler 7. Condensate from intercooler 7 is returned to liquid space 2 by drain leg 8, having in its sealing loop 9.

Vapors and gases uncondensed in intercooler 7 are removed by steam jet 10. On drain leg 8 we have provided cooler 11, supplied with cold water through line 12, which water departs through line 13. Although the preferred form shown here is a body surrounding the drain seal 9, it is also possible to cool the liquid by any of the usual forms of tubular heat exchange devices placed at any point in the run of line 8 between intercooler 7 and liquid space 2. The form shown is the simplest, and probably the cheapest, though possibly not the most efficient.

We claim:

1. In apparatus for distillation and fractionation under high vacuum making use of a vacuum condenser and multistage steam jet vacuum pumps with intercoolers from which condensate is returned to the vacuum condensate collection zone, means for cooling such intercooler condensate below the temperature at which its vapor pressure is equal to the pressure existing in the vacuum condensate collection space.

2. In apparatus for distillation and fractionation under high vacuum making use of a vacuum condenser and multistage steam jet vacuum pumps with intercoolers from which condensate is returned to the vacuum condensate collection zone, means for cooling such intercooler condensate below the temperature at which its vapor pressure is equal to the pressure existing in the vacuum condensate collection space comprising an indirect heat exchange device.

3. In the operation of distilling and fractionating under high vacuum, when making use of a vacuum condenser and multistage steam jet vacuum pumps with intercoolers from which condensate is returned to the vacuum condensate collection zone, the step of reducing the vapor pressure of such returned intercooler condensate to a value less than the pressure obtaining in such vacuum condensate collection zone before admitting it thereto.

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